

California Environmental Protection Agency



PROPOSED

Permeation Test Procedure

Test Method 513

**DETERMINATION OF PERMEATION RATES FOR SPILL-
PROOF SYSTEMS**

Adopted: _____

**California Environmental Protection Agency
Air Resources Board**

Permeation Test Procedure

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**Determination of Permeation Rates For
Spill-Proof Systems**

1 APPLICABILITY

For the purpose of this procedure, the term "ARB" refers to the State of California Air Resources Board, and the term "ARB Executive Officer" refers to the Executive Officer of the ARB or his or her authorized representative or designate.

This procedure is used to determine the permeation rate of spill-proof systems with non-metallic portable fuel containers. It is applicable in all cases where a spill-proof system is sold, supplied, offered for sale, or manufactured for use in the state of California

This test procedure involves the use of flammable materials and operations and should only be used by or under the supervision of those familiar and experienced in the use of such materials and operations. Appropriate safety precautions should be observed at all times while performing this test procedure.

2 PRINCIPAL AND SUMMARY OF TEST PROCEDURE

Portable fuel containers are filled with gasoline and allowed to precondition at ambient temperature and pressure for a minimum of four weeks. The portable fuel containers are then emptied, blown dry and immediately refilled with Phase II California Reformulated Certification (CERT) Fuel. The containers are then plugged and sealed with an impermeable epoxy sealant. After the sealant has cured the containers are weighed and subjected to a 24-hour variable temperature profile. The containers are then re-weighed and the weight loss in grams is calculated.

3 BIASES AND INTERFERENCES

Portable fuel containers incorrectly sealed will emit evaporative emissions, which can effect

the final weight loss calculations.

The balance used to determine the weight loss between 24-hour variable temperature profiles must be of sufficient capacity to accurately weigh large volume portable fuel containers filled to their nominal capacity with CERT fuel.

4 SENSITIVITY, RANGE, AND PRECISION

Range of measurement of filled portable fuel containers is approximately 3,000 grams to 26,000 grams; upper range depends on the volume of the portable fuel container.

5 EQUIPMENT

5.1 Brass cap or plug

Use a threaded brass cap or plug of sufficient size to completely cover or plug the opening of the portable fuel container.

5.2 High capacity toploading balance

Use a high capacity balance capable of a maximum weight measurement of not less than 32,000 grams with a minimum readability of 1 gram and a reproducibility of $\leq \pm 0.2$ grams.

5.3 Sealed Housing for Evaporative Determination (SHED)

Use either a fixed or variable volume enclosure as specified in 40 CFR, Chapter 1, Part 86, Section 1207-96 with a temperature conditioning system capable of controlling the internal enclosure air temperature with an instantaneous tolerance of ± 3.0 °F of the nominal temperature versus time profile throughout the test, and an average tolerance of 2.0 °F over the duration of the test.

6 CALIBRATION PROCEDURE

The high capacity toploading balance shall be calibrated prior to use per the manufacturer

specifications.

7 DURABILITY PROCEDURE

This section is reserved for future specification.

8 PRECONDITIONING PROCEDURE

Fill the portable fuel container of the spill-proof system to its nominal capacity with gasoline and firmly attach either the spill-proof spout or an appropriate plug or cap. Place the portable fuel container in a suitable vented enclosure. Record the preconditioning start date on the field data sheet (see figure 2). The portable fuel container shall remain undisturbed for a period of not less than four weeks.

9 SEALING PROCEDURE

- (1) After preconditioning, remove the portable fuel container from the enclosure to a well-ventilated area. Record the preconditioning end date on the field data sheet. Remove the cap, plug, or spill-proof spout and empty the portable fuel container. The portable fuel container must not remain empty for more than fifteen minutes. Quickly dry the interior of the portable fuel container with compressed air. Immediately refill the portable fuel container to its nominal capacity with CERT fuel. Use an appropriate threaded cap or plug to completely seal the opening of the portable fuel container. Coat threaded areas liberally with an impermeable epoxy sealant. Firmly attach the threaded cap or plug. (If a plug is used it may be necessary to install a cover cap or band clamp around the opening at the level of the plug. Container swelling during the test may cause the opening to distort and separate from the plug.) Apply an overcoat of sealant around the entire opening including the cap or exposed area of the plug. Secure the sealed portable fuel container and allow sufficient time for the sealant to cure before proceeding with the remainder of the sealing procedure.
- (2) After allowing sufficient time for the curing of all sealant on or around the closure, heat the sealed portable fuel container until positive pressure (container swelling) is observed. This could be accomplished in several ways. One method is to place the portable fuel container in a well-ventilated area exposed to direct sunlight for two to four hours. If positive pressure (container swelling) is not observed after heating, the closure or portable fuel container is leaking. Reevaluate the selection of treaded cap or plug and use good engineering practices to correct the problem. Once positive pressure is observed test the integrity of the closure and sealant by completely immersing the portable fuel container in a water bath for a period of two minutes. Select a water bath large enough to completely cover the portable fuel container plus six inches. Place the portable fuel container upright

in the water bath making sure to position it so that no fuel is in contact with the closure. To accomplish this it may be necessary to tilt the portable fuel container back slightly so that the closure is the highest point while holding it under water. Observe the portable fuel container and the closure for any leaks. Leak points will be visible as a bubble or stream of bubbles while immersed in the water bath. Identify and mark any leak points. If leaks are observed remove and dry the portable fuel container and repair all leaks. Continue this process until no leaks are observed

10 TEST PROCEDURE

- (1) Make sure that the exterior surface of the sealed portable fuel container is clean, dry, and free of dirt and debris. Carefully place the sealed portable fuel container on the high capacity balance. Record the initial weight (W_i), date, and start time on the field data sheet.
- (2) Immediately place the sealed portable fuel container in the SHED. Begin the 24-hour variable temperature profile (see figure 1). If more than one hour elapses between the time the sealed portable fuel container was weighed and the initiation of the variable temperature profile, the sealed portable fuel container must be re-weighed before initiating the 24-hour variable temperature profile.
- (3) At the conclusion of the 24-hour variable temperature profile immediately remove the sealed portable fuel container from the SHED and ensure that the exterior surface is clean, dry, and free of dirt and debris. Carefully place the sealed portable fuel container on the high capacity balance. Record the final weight (W_f), date, and end time on the field data sheet. Calculate the difference between the initial weight (W_i) and the final weight (W_f). This is the weight loss (W_l) due to permeation. Record the weight loss (W_l) on the field data sheet. If more than one hour elapses between the conclusion of the 24-hour variable temperature profile and the final weighing of the sealed portable fuel container, the final weight is invalid and should not be used in future calculations. If this occurs, the test procedure must be reinitiated.
- (4) Repeat this process until the weight loss (W_l) from five consecutive 24-hour cycles displays a standard deviation of ≤ 0.25 grams.
- (5) When sufficient weight loss data have been collected, move the sealed portable fuel container to a well-ventilated area. Place the sealed portable fuel container on a clean sheet of paper. Carefully remove the threaded plug or cap allowing the loose sealant to fall on the paper. Drain the contents of the portable fuel container into an appropriate receptacle through a finely screened funnel. Dry the interior of the portable fuel container with compressed air. Check the screened funnel for any sealant that may have fallen off

the portable fuel container during draining. Place any particles of sealant found in the screened funnel on the paper and allow them to air dry. Collect the particles of sealant from the paper, the cap or plug, and the portable fuel container and place them on the high capacity balance. Record this weight on the field data sheet as the tare weight (W_t).

11 QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

This section is reserved for future specification.

12 RECORDING DATA

Record data on a form similar to the one shown in Figure 2.

13 CALCULATING RESULTS

The weight loss in grams is calculated for each 24-hour cycle as follows:

$$Wl = Wi - Wf$$

Where:

- Wl = The weight loss in grams in one day
- Wi = The initial weight of the portable fuel container in grams
- Wf = The final weight of the portable fuel container in grams after one day

The standard deviation of five consecutive diurnal cycles is calculated as follows:

$$SDV = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n - 1}}$$

Where:

- SDV = Standard Deviation
- n = number of samples
- X_i = i th weight loss in grams
- \bar{X} = mean of weight losses in grams

The permeation rate in grams/gallon/day for each 24-hour cycle is calculated as follows:

$$P = \overline{\left(\frac{-}{d} \right)}$$

Where:

<i>P</i>	= Permeation rate in grams/gallon/day
<i>Wl</i>	= Weight loss in grams
<i>Wi</i>	= Initial weight in grams
<i>Wt</i>	= Tare weight in grams
<i>d</i>	= Density of CERT fuel in grams/gallon

14 REPORTING RESULTS

After calculating the permeation rate for each 24-hour cycle, an average of the five consecutive rates selected is calculated to determine the final permeation rate in grams/gallon/day.

15 ALTERNATIVE TEST PROCEDURES

Test procedures, other than specified above, shall only be used if prior written approval is obtained from the ARB Executive Officer. In order to secure the ARB Executive Officer's approval of an alternative test procedure, the applicant is responsible for demonstrating to the ARB Executive Officer's satisfaction that the alternative test procedure is equivalent to this test procedure.

- (1) Such approval shall be granted on a case-by-case basis only.
- (2) Documentation of any such approvals, demonstrations, and approvals shall be maintained by the ARB Executive Officer and shall be made available upon request.

16 REFERENCES

This section is reserved for future specification.

17 FIGURES

Figure 1. 24-Hour Variable Temperature Profile

Figure 2. Field Data Sheet

Figure 1. 24-Hour Variable Temperature Profile

1 Day / 24 Hour / 1440 Minute Variable Temperature Profile

HOUR	MINUTE	ET / MIN	TEMP °F
0	0	1440	65.0
1	60	1380	66.6
2	120	1320	72.6
3	180	1260	80.3
4	240	1200	86.1
5	300	1140	90.6
6	360	1080	94.6
7	420	1020	98.1
8	480	960	101.2
9	540	900	103.4
10	600	840	104.9
11	660	780	105.0
12	720	720	104.2
13	780	660	101.1
14	840	600	95.3
15	900	540	88.8
16	960	480	84.4
17	1020	420	80.8
18	1080	360	77.8
19	1140	300	75.3
20	1200	240	72.0
21	1260	180	70.0
22	1320	120	68.2
23	1380	60	66.5
24	1440	0	65.0

Figure 1. Field Data Sheet

Spill-Proof System Mfg. _____
 Container Volume _____
 Container I.D. _____
 Inspector _____
 Water Bath Test (pass/fail) _____
 Tare Weight (*W_t*) (grams) _____

DATE START END		TIME	INITIAL WEIGHT <i>W_i</i> (grams)	FINAL WEIGHT <i>W_f</i> (grams)	WEIGHT LOSS <i>W_l</i> (grams)